RESEARCH AND EXPLORATORY DEVELOPMENT ON THE FABRICATION AND CHARACTERIZATION OF FIBER REINFORCED LIGHT METAL ALLOY COMPOSITES

FINAL REPORT

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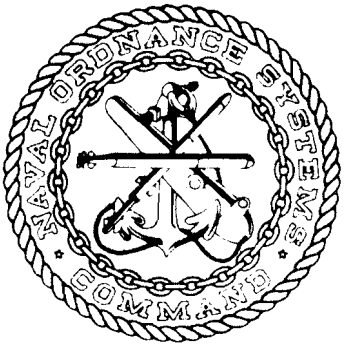

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FOREWORD

Grateful acknowledgement is made of the continued guidance and helpfulness of Mr. Marlin Kinna and Mr. Sol Matesky, Naval Ordnance Systems Command, during the course of this program.

The authors also wish to express appreciation to their colleagues at ARTECH CORP., particularly Mrs. Leah Loy and Messrs. Russell Rowe and Orville VanBlaricon who participated in the fabrication and testing reported herein.

ABSTRACT

Discontinuous β-SiC whiskers and chopped C filaments were used as a reinforcement for strengthening 7178 and 2024 Al alloys. The C fibers were coated with Cu to improve wetting. Tensile properties of a 25 v/o β-SiC 2024 Al composite were increased three times over the matrix strength at room temperature and ten times at 800°F. Tensile properties in the transverse direction were at least equal to the matrix strength at temperatures up to 800°F.

Ti-10V composites reinforced with SiC filaments were consolidated by activated pressure sintering. The samples produced were not fully dense. Consolidation temperature or time at temperature could not be increased since the reaction between the matrix and filaments would be too severe. Work on this system was terminated.

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SECTION I

INTRODUCTION

The problem of providing materials that can be highly stressed without the danger of catastrophic failure has led to the development of composites. Composite materials are an effective way to combine materials of high strength and/or elastic modulus per unit weight, which typically are brittle, with more ductile materials so that failures tend to be limited and localized. Such combinations of high strength and comparatively low density are suitable for light weight structures and equipment to improve naval systems, such as deep submersibles, by reducing weight.

ARTECH has for the past six years been actively engaged in composite research and development. The work mainly has been concerned with the development of suitable fabrication techniques for composite materials and the subsequent evaluation of the materials so produced.

The objective of this program has been the development of light metal matrix composites with transverse properties of the composites equal to those of the matrix. Applying the knowledge and techniques developed in NOSC Contract N00017-71-C-4404 and NASC Contract N00019-70-C-0205, discontinuous β-SiC and chopped C fiber composites have been produced by liquid phase hot pressing. The 25 v/o β-SiC/2024 Al composites produced and tested during the program have met this objective. In addition, SiC filament/Ti-10 v composites have been studied.

SECTION II

EXPERIMENTAL PROCEDURES

1. Materials

a. Metal Matrices

The matrices used in this program included titanium and aluminum alloys. Titanium-vanadium powder, -400 mesh, containing ten weight percent vanadium was produced by Oremet Corp, Albany, Oregon. The 2024 and 7178 Al alloy atomized powders, obtained from the Reynolds Metals Company, Richmond, Virginia were sieved and the -400 mesh fraction was utilized for the composite matrices.

b. Reinforcing Fibers

Silicon carbide filaments of 0.004 in. diameter, obtained from Thompson CSF, Orlay, France, were used as the reinforcement in the Ti-V matrix composites. Modmor Type 1 graphite fibers, chopped to 1/4 in. average length, from Fothergill & Harvey Limited, Littleborough, Lanes., England, were utilized as reinforcement in Al alloy composites. β-SiC whiskers, purchased from Lonza Limited, Basel, Switzerland, were also used as reinforcement in the Al alloy composites.

2. Composite Preparation and Consolidation

a. β-SiC Reinforced Al Alloy Composites

The β-SiC whiskers most recently obtained from Lonza Ltd. were markedly superior to domestic whiskers in usable whisker content, but not sufficiently free of nonwhisker material or completely dispersed to be directly combinable with the aluminum alloy powder for the fabrication of composite specimens. The undesirable material, fine black platelets and small pieces of undispersed, as-grown whisker mat, were considered sufficient in quantity to impair the quality of fabricated specimens.

Processing of several small batches of the as-received whisker material, in the cleaning and classifying apparatus described in the final report (1) to NASC, indicated that the fine black platelets could be completely removed without major effort. However, the small pieces

of undispersed, as-grown whisker mat could not be removed as they easily passed through or broke down in size and were forced through the classifying screens.

Attempts were made to disperse this material by ultrasonically blasting small batches of as-received whisker material in water with a Sonabond immersible probe. Some dispersion occurred, and several surfactants were added to the batches to further aid dispersion. A commercial laboratory glassware detergent containing sodium hexametaphosphate appeared to yield the best results. Complete dispersion was obtained with twenty minutes of the ultrasonic treatment.

The remaining as-received whisker material was processed by the following procedure:

1. Ultrasonically disperse whisker material in warm water containing approximately 0.1% Sparkleen detergent.

2. Process dispersed material in cleaning and classifying apparatus using No. 400 screen basket.

3. Process retained whiskers from No. 400 screen basket with No. 50 screen basket and collect whiskers washed through basket.

4. Process retained whiskers from No. 50 screen basket with No. 24 screen basket and collect whiskers washed through basket.

5. Collect whiskers retained in No. 24 screen basket.

The material washed through the self-cleaning No. 400 screen basket, consisting of powder and ultrafine whiskers, was considered too unproductive for further processing. Of the recovered whiskers (approximately half of the starting material), only those retained in the No. 24 screen basket (10% of the starting material) were not used in composites because of their tendency to intertwine and form strands.

All composites, regardless of their final alignment, were initially prepared as green bodies with random X-Y alignment in order to optimize the distribution of the whiskers and powder. To produce unidirectional alignment

the dried green body was infiltrated with melted camphene that had been previously distilled. After the camphene had solidified, the billets were extruded into 1/8-in. square bars to align the whiskers. The bars were cooled to retard evaporation of the camphene, straightened, and cut into appropriate lengths. The cut lengths were stacked in a Teflon lined die with a cavity 1/4 in. by 1-5/8 in. in cross section and a single plunger. The whisker direction was parallel to either the 1/4 or the 1-5/8 in. dimension depending on the orientation desired in the final composite. The camphene was removed by placing the assembled die on the heating/cooling platen of a hand-operated hydraulic press, with the open end on a layer of absorbent paper, and applying a force just sufficient to keep the bottom of the stacked bars in contact with the heated absorbent surface.

All whisker composites were consolidated by liquid phase hot pressing in a die lined with graphite (Poco HPD-1). The die, shown in figure 1, was fitted with metal expansion seals to retain the liquid formed during hot pressing. In most cases consolidation parameters for the two matrices were:

Matrix	Temperature, °C	Pressure, psi
2024 Al	600	3000
7178 Al	532	3000

b. SiC Filament Reinforced Ti-10V Alloy Composites

During the previous investigation, (2) initial results on composite specimens containing SiC fibers axially aligned in a Ti-10V alloy matrix appeared promising in that readily achievable fabrication parameters produced desirable mechanical properties. The process included activated sintering of the hydrided matrix alloy. Decomposition of the hydride powder and consolidation of the composite in vacuum appeared to be complete after hot pressing at 800°C and 4000 psi for 3 hrs.

Starting with these parameters, work was begun during the first quarter of the present contract to prepare a sufficient number of composite specimens of higher SiC fiber volume fraction for evaluation of the composite system. Three specimens were prepared by the technique described in the previous final report in an effort to evaluate and eliminate the deficiencies of the technique. Two specimens contained three layers of fibers and the third contained six layers. All specimens were sectioned into eight pieces for metallographic examination. Two basic problems appeared to limit

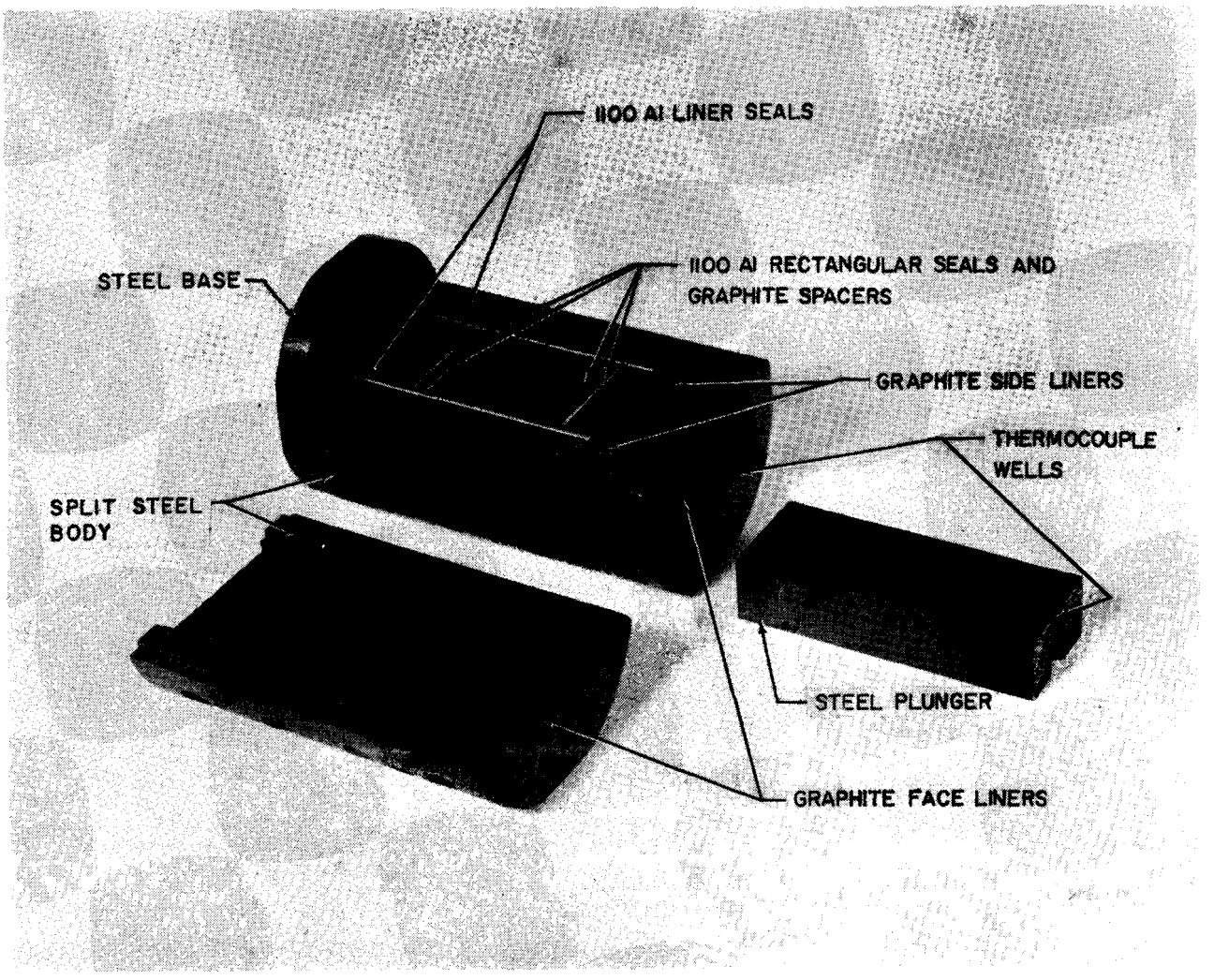

Figure 1. Graphite die used to consolidate composites by liquid phase hot pressing.

the effectiveness of the reinforcing capability of the SiC fibers. The layer of matrix powder varied in thickness along the length of the specimen, and the fibers were displaced, probably during the initial stage of camphene removal.

To solve the problem of powder uniformity, several alternative methods of preparing the camphene/hydride powder extrusion billet were examined. The most uniform distribution of powder in the extruded strip was obtained by allowing the hydride powder to settle completely before the mixture cooled and the camphene solidified.

The ideal way of maintaining the desired fiber spacing would be to lay up the fibers in grooved strips of the hydride. In a cursory experiment, hydride powder was hot pressed at 600°C and 4000 psi for 1 hr and yielded a strip that was readily released from the graphite die, not friable, and sufficiently rigid for handling. As strips less than 0.020 in. thick are necessary to prepare specimens of adequate fiber content, tests were made to determine whether thin strips of uniform thickness could be prepared. Three strips of each of three proportions were hot pressed from extruded camphene/hydride powder strips to as-pressed thicknesses of 0.007 to 0.025 in.

The camphene/hydride powder proportions having been established, steel platens were fabricated to form seven grooves centrally located on one face of the strip and eight grooves on the other. The first hot pressed strip was found to be mechanically locked to the platens, but was removed without damage. The second strip broke during removal, damaging the grooves in the platens. Platens made from Poco HPD-1 graphite produced the desired results, the strips separating freely. Fourteen hydride strips, 0.020 in., were hot pressed with these platens at 600°C and 4000 psi for 1 hr for eventual use in composite specimens.

Several composite samples, with fibers cut to the length of the die cavity and 0.005 in. smaller than the die cavity, were fabricated for metallographic evaluation. The composite specimens were consolidated at 800°C and 4000 psi for 3 hr. Five of these consisted of single grooved strips, and one of two grooved strips. In each case, the composite was assembled by positioning the SiC fibers in the grooves and retaining them with melted camphene. This assembly was then sandwiched between strips of camphene/hydride powder which was in turn sandwiched between 1/32-in. strips of Ti-75A titanium alloy.

With the assurance that the fabrication process was not responsible for fracturing fibers, six test samples were prepared by hot pressing at 4000 psi and 800°C for three hours.

The three test samples without fibers were composed of a grooved strip of Ti-10V hydrided powder previously hot pressed at 600°C and strips of camphene, containing the hydrided powder, at the top and bottom of each grooved strip. These samples were consolidated by bringing the ram into contact, allowing the pressure to rise as the temperature increased, and applying full pressure at the consolidating temperature.

The three test samples containing the fibers were prepared in the same way, the fibers being placed in the grooved strips and held in place with solidified camphene. Backup strips of titanium RS 70 alloy were placed at the top and bottom of the powder strip in each sample. Consolidation of these composite samples was identical with that of the unreinforced samples except that a low pressure was initially applied and then the pressure was allowed to increase as the temperature increased, with full pressure being applied at the consolidating temperature. The initial pressure was not applied to the third composite sample.

Two additional composite samples were prepared using two grooved strips in each. The consolidation parameters were identical for these two composite samples except that the initial consolidating pressure was applied to only one.

c. Copper Coated C Filament Reinforced Al Alloy Composites

To reduce the effects of surface impurities on the chopped graphite filaments, the fibers were washed three times in boiling water and fired in nitrogen at 900°F. Following cleaning, the graphite filaments were coated with an electroless Cu plate as follows:

(1) Approximately one-third of a gram of fibers was immersed in one liter of HCl solution (3 parts H_2O, 1 part HCl) for three minutes.

(2) Water rinse.

(3) The fibers were immersed in one liter of Shipley Cuposit Catalyst 6F for 3 minutes.

(4) Water rinse.

(5) The fibers were immersed in one liter of a Shipley Cuposit Accelerator 19 solution (1 part accelerator to 5 parts H_2O) for 5 minutes.

(6) The fibers were coated for 10 minutes at 75°F in Shipley Copper Mix 328.

(7) The fibers were water rinsed and oven dried for 10 minutes.

The Cu coated chopped fibers were prepared and consolidated by camphene infiliation, extrusion and liquid phase hot pressing as described in the previous year's work[2].

SECTION III

TESTING PROCEDURES

1. Mechanical Testing

Tensile testing was performed on an Instron machine floor model TTC. A chart speed of 1/2 in./min was employed. A strain rate of 0.02 in./in./min was used in the majority of cases. For the room temperature specimens, the elastic modulus was determined by use of a 10%, 1/2-in. strain gage extensometer in conjunction with an X-Y chart recorder. For the elevated temperature specimens, the elastic modulus was determined by using a Baldwin strain follower in conjunction with an X-Y chart recorder.

Longitudinal and transverse composite and matrix tensile specimens were tested at room temperature, 400, 600 and 800°F. The subsize tensile specimens tested are shown in figure 2. The elevated temperature tests were made using a clam shell furnace controlled to ± 10°F. Samples were soaked for 15 minutes at the indicated temperatures before testing was begun.

2. Metallography

Standard mounting and polishing techniques were used to prepare composite specimens for metallographic examination. A hard mounting medium, Buehler No. 20-8130AB epoxide compound (oven-cured), was preferred in order to reduce rounding of the specimen edges.

Grinding with Varsol on SiC papers through 600 grit was used to prepare the specimens for the polishing operation. Polishing with diamond abrasives of -15, -6, -3, and -1 micron grit sizes was found to be most effective regardless of the fiber content of the specimen. Polishing papers, giving a comparatively hard lap surface, were most effective in minimizing relief at the fiber-matrix interfaces. With the titanium composite specimens, polishing was done with chromic acid-levigated alumina slurries on low nap cotton cloth. Final polishing was done using chromic acid-levigated 0.05 micron alumina on Microcloth.

Standard etchants for aluminum and titanium alloys were used to outline the fibers, bring out the grain boundaries, and remove disturbed metal at fiber-matrix interfaces.

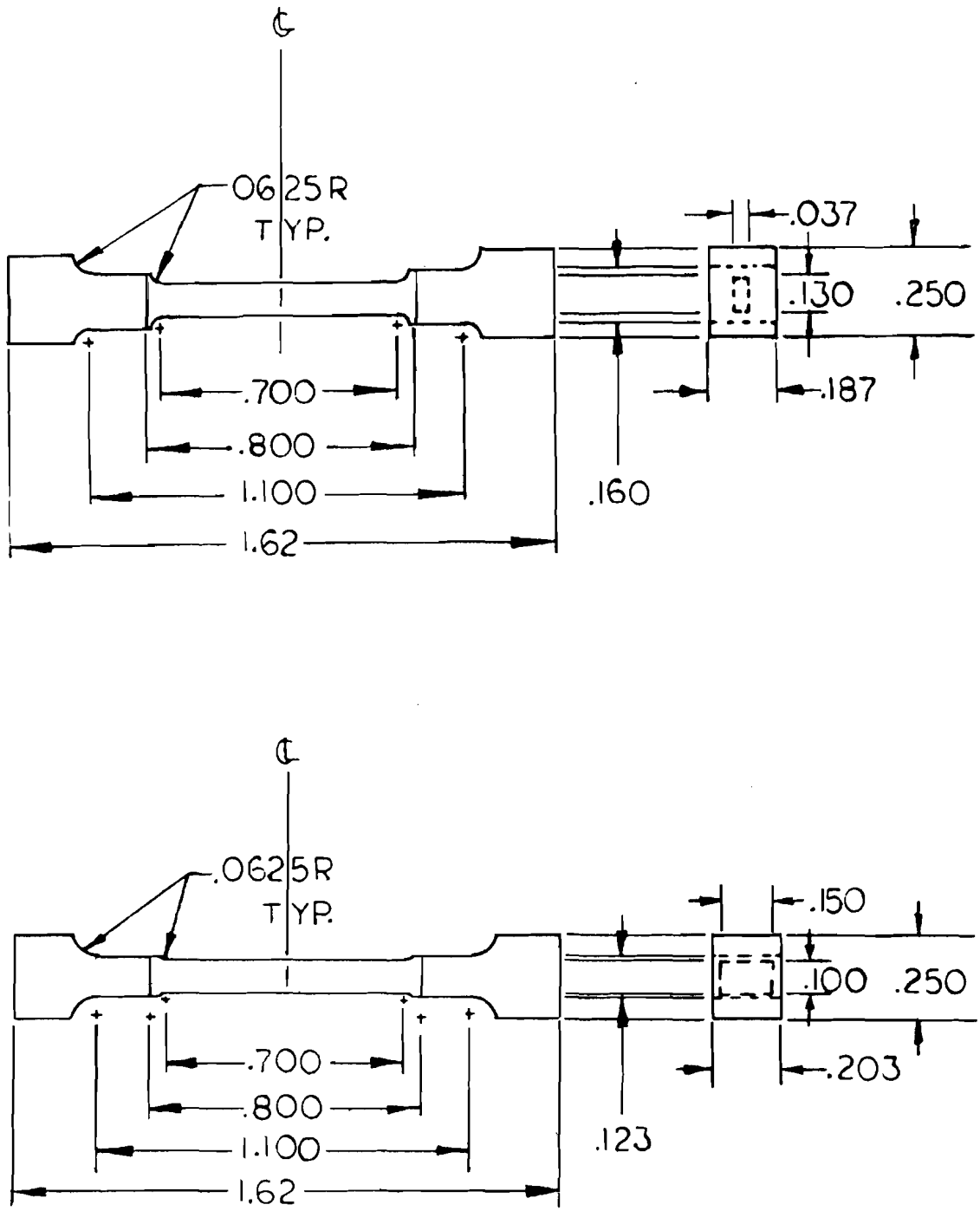

Figure 2. Subsize tensile specimen configurations employed in tensile testing Ti-10V/SiC composites (top) and aligned 2024Al/25 v/o β-SiC composites (bottom).

Extraction of SiC filaments to assess filament damage from the consolidation process was conducted. The digestion in $HF-HNO_3$ was observed periodically at low magnification after the initial violent reaction had subsided. As the fibers lying parallel to the surface became visible, cracks were not visible at first. As the etching proceeded, the fibers were observed to bow out of the surface and eventually crack emitting audible sounds. After this observation several additional specimens were prepared and potted in epoxy resin so that etching could proceed only from the ends of the specimens. Although the reaction was significantly slower because of the smaller exposed surface, etching in this manner was found to produce unfractured fibers. The fibers used in these specimens were cut initially to fit so closely in the hot press die that elongation of the fiber was restricted to less than 1% during the consolidation. Samples containing filaments which were cut to fit loosely in the die were observed to be fractured in the consolidation process.

3. Density

The specimens were ultrasonically cleaned in a series of reagent grade solvents prior to determining the density. The first solvent, benzene, was followed by acetone, then deionized water, and finally ethanol, followed by oven drying. Several specimens, prior to cleaning, required fine grinding to remove graphite adhering to the surface.

The displacement method was used to determine the densities of all specimens. Isopropyl alcohol was used as the immersion liquid because its low surface tension permitted penetration of surface porosity, which could be detected by the displacement of air bubbles or by a slow increase in weight.

SECTION IV

RESULTS AND DISCUSSIONS

1. β-SiC Reinforced Al Alloy Composites

Of the sixty-five pressings made for the testing program, fifty-seven were successfully consolidated using the 2024 Al alloy as the matrix. The remainder represented attempts of rather limited success to produce samples initially containing 40% whiskers aligned in a 7178 Al alloy matrix.

7178 Al Alloy Matrix

Previous attempts to produce aligned high volume percent whisker composites were unsuccessful primarily because of the great difference in size between the whiskers (1-3 μ dia. x 100 - 1000 μ long) and matrix powder (-325). The distribution of this size matrix powder, comprised mainly of particles slightly less than 325 mesh, in the massive number of small whiskers was considered insufficient to form a continuous liquid envelope around the whiskers when pressing at low pressures in the liquid and solid region of the matrix. Consolidation of these composites with the matrix above the liquidus temperature was unsuccessful because of the inability of the hot-pressing die to sufficiently retain the liquid.

The attempts to consolidate high volume percent whisker composites on this program were primarily based upon the use of ultrafine matrix powder and the demonstrated ability in the present hot-press die to significantly reduce or prevent the loss of liquid during the consolidation of the 2024 Al alloy/β-SiC whisker composites.

The matrix powder used for the 7178 alloy composites was obtained by passing -325 mesh powder through a 400 mesh screen. Microscopic examination of the -400 mesh fraction indicated that the major portion of it was much less than 400 mesh and further separation into finer sizes was considered unwarranted. Consolidation of this powder into matrix samples without whiskers was entirely successful with densities being greater than 99% of theoretical, thus proving the reliability of the hot-press die for this alloy composition. However, all attempts to consolidate a composite with 40% whiskers resulted in samples with low as-pressed densities.

By increasing the liquid content from 30 to 100 percent, the resulting densities were only increased to approximately 85% of theoretical. An attempt was made to use approximately 25 volume percent of whiskers initially and increase content to about 30 volume percent by allowing a portion of the liquid to be transferred to a reservoir during consolidation. Although the resulting composite produced by this modification appeared better than previous attempts in terms of density, sufficient funds and time were not available to fabricate the additional composite samples necessary to fully determine the potential of the technique.

The inability to fabricate completely dense whisker composites over 35 v/o may well be associated with micro-perturbations in the alignment of the whiskers with respect to each other. The presence of whiskers misaligned by even the smallest of angles created a larger number of voids. When these are filled with matrix, an insufficient amount remains available for full coverage of all whisker surfaces. Hence, unless perfect alignment is attained or imposed during consolidation the fabrication of these composites appears in doubt.

2024 Al Alloy Matrix

As indicated above, fifty-seven samples were successfully consolidated using 2024 Al alloy for the matrix. Of these samples seventeen each contained whiskers longitudinally and tranversely aligned for determination of their mechanical properties at room and elevated temperatures. The remainder constituted matrix samples without whiskers for comparison with the determined properties of the composites. Only one tensile specimen could be machined from each sample, and only those samples with a density greater than 97% of theoretical were selected for testing. Prior to machining all samples were heat treated by solution annealing at 920°F ± 5°F for 7 hours followed by quenching in ice water and artificial aging at 365°F ± 5°F for 8 hours.

The results determined from the room and elevated temperature tensile tests are shown in Tables 1 through III and graphically presented in figures 3 through 6. The results show significant reinforcement of the 2024 Al alloy matrix achieved by the incorporation of 25 v/o β-SiC whiskers. All measured properties including proportional limit, tensile yield strength, ultimate tensile strength, and modulus of elasticity were significantly increased, particularly for the specimens in which the whiskers had been longitudinally aligned. For these specimens the ultimate tensile strength was improved by nearly a factor

TABLE I

SHORT-TIME ELEVATED TEMPERATURE
TENSILE PROPERTIES OF A 2024 A1 TYPE ALLOY
CONSOLIDATED BY LIQUID PHASE HOT PRESSING

Specimen Number	Density g/cm^3	Test Temperature ^oF	Cross Sectional Area $in.^2$	Tensile Properties				Fracture Location
				Proportional Limit kpsi	Yield Strength kpsi (a)	Ultimate Strength kpsi	Elastic Modulus Mpsi	
4B	2.77	75	0.0154	(b)	38.0	55.2	9.1	A
5B	2.77	75	0.0149	(b)	39.7	49.0	11.2	B
10B	2.76	75	0.0148	30.4	38.8	52.7	10.1	A
12B	2.78	75	0.0149	32.2	41.6	47.7	10.4	B
14B	2.77	75	0.0146	35.6	43.3	47.0	11.2	B
7B	2.77	400	0.0152	(c)	(c)	32.0	(c)	A
11B	2.76	400	0.0150	22.7	30.3	30.9	9.8	B
13B	2.77	400	0.0149	(d)	(d)	24.4	(d)	B
15B	2.76	400	0.0151	(c)	(c)	32.6	(c)	A
35B	(b)	600	0.0151	3.6	7.9	9.0	3.9	B
37B	(b)	600	0.0153	5.8	8.8	9.9	5.4	B
50B	2.74	600	0.0154	4.0	7.3	8.8	3.9	B
32B	2.77	800	0.0150	2.3	4.1	4.3	4.4	B
34B	(b)	800	0.0157	2.2	3.9	4.4	2.5	B
36B	(b)	800	0.0154	2.6	4.2	4.3	3.7	B

(a) 0.2% offset
(b) not determined
(c) UTS specimen only
(d) curve unsuitable for property determination

A - in gage length
B - at knife edge

TABLE II

SHORT-TIME ELEVATED TEMPERATURE
LONGITUDINAL TENSILE PROPERTIES OF ALIGNED
2024 Al/25 v/o β-SiC COMPOSITES CONSOLIDATED
BY LIQUID PHASE HOT PRESSING

Specimen Number	Density g/cm^3	Test Temperature ^oF	Cross Sectional Area $in.^2$	Tensile Properties				Fracture Location
				Proportional Limit kpsi	Yield Strength kpsi (a)	Ultimate Strength kpsi	Elastic Modulus Mpsi	
22L	2.88	75	0.0149	101(b)	111	152	21.8	C
47L	2.80	75	0.0151	66.2	119	153	23.8	C
53L	2.85	75	0.0153	81.7	137	155	25.6	C
23L	2.87	400	0.0147	(c)	(c)	93.2	(c)	C
25L	2.88	400	0.0153	46.7	126	128	26.5	B
24L	2.89	400	0.0150	(c)	(c)	128	(c)	C
44L	2.82	400	0.0156	57.7	88.4	111	22.8	C
18L	2.86	600	0.0134	22.4	51.9	61.6	18.3	B
54L	2.85	600	0.0155	32.2	67.8	75.8	16.7	A
45L	2.85	800	0.0152	18.5	36.8	45.1	16.1	C
49L	2.90	800	0.0152	23.1	38.9	42.8	17.2	D
52L	2.85	800	0.0154	(d)	(d)	36.7	(d)	C

(a) 0.2% offset
(b) after initially straining to 0.27%
(c) UTS specimen only
(d) curve unsuitable for property determination

A - in gage length
B - at knife edge
C - near radius
D - in top grip

TABLE III

SHORT-TIME ELEVATED TEMPERATURE
TRANSVERSE TENSILE PROPERTIES OF ALIGNED
2024 Al/25 v/o β-SiC COMPOSITES CONSOLIDATED
BY LIQUID PHASE HOT PRESSING

Specimen Number	Density g/cm^3	Test Temperature ^oF	Cross Sectional Area $in.^2$	Tensile Properties				Fracture Location
				Proportional Limit kpsi	Yield Strength kpsi (a)	Ultimate Strength kpsi	Elastic Modulus Mpsi	
9T	2.85	75	0.0152	24.3	(b)	46.1	17.0	C
29T	2.88	75	0.0152	29.5	46.4	51.8	14.7	A
38T	2.87	75	0.0138	39.8	51.2	56.5	16.2	C
6T	2.87	400	0.0151	14.6	25.9	34.4	13.2	B
27T	2.88	400	0.0148	(c)	(c)	35.7	(c)	C
28T	2.87	400	0.0151	(c)	(c)	34.4	(c)	C
39T	2.88	400	0.0153	17.4	25.8	29.8	18.6	A
19T	2.85	600	0.0150	(c)	(c)	13.0	(c)	C
20T	2.85	600	0.0150	(c)	(c)	13.9	(c)	C
26T	2.88	600	0.0152	6.6	10.3	12.8	10.1	B
46T	2.87	600	0.0154	9.7	11.5	13.8	10.0	A
57T	2.88	600	0.0151	7.6	11.2	13.2	10.8	A
17T	2.83	800	0.0125	3.0	5.2	6.0	2.7	B
30T	2.87	800	0.0151	(c)	(c)	6.1	(c)	C

(a) 0.2% offset
(b) failed at less than 0.2% strain
(c) UTS specimen only

A - in gage length
B - at knife edge
C - near radius

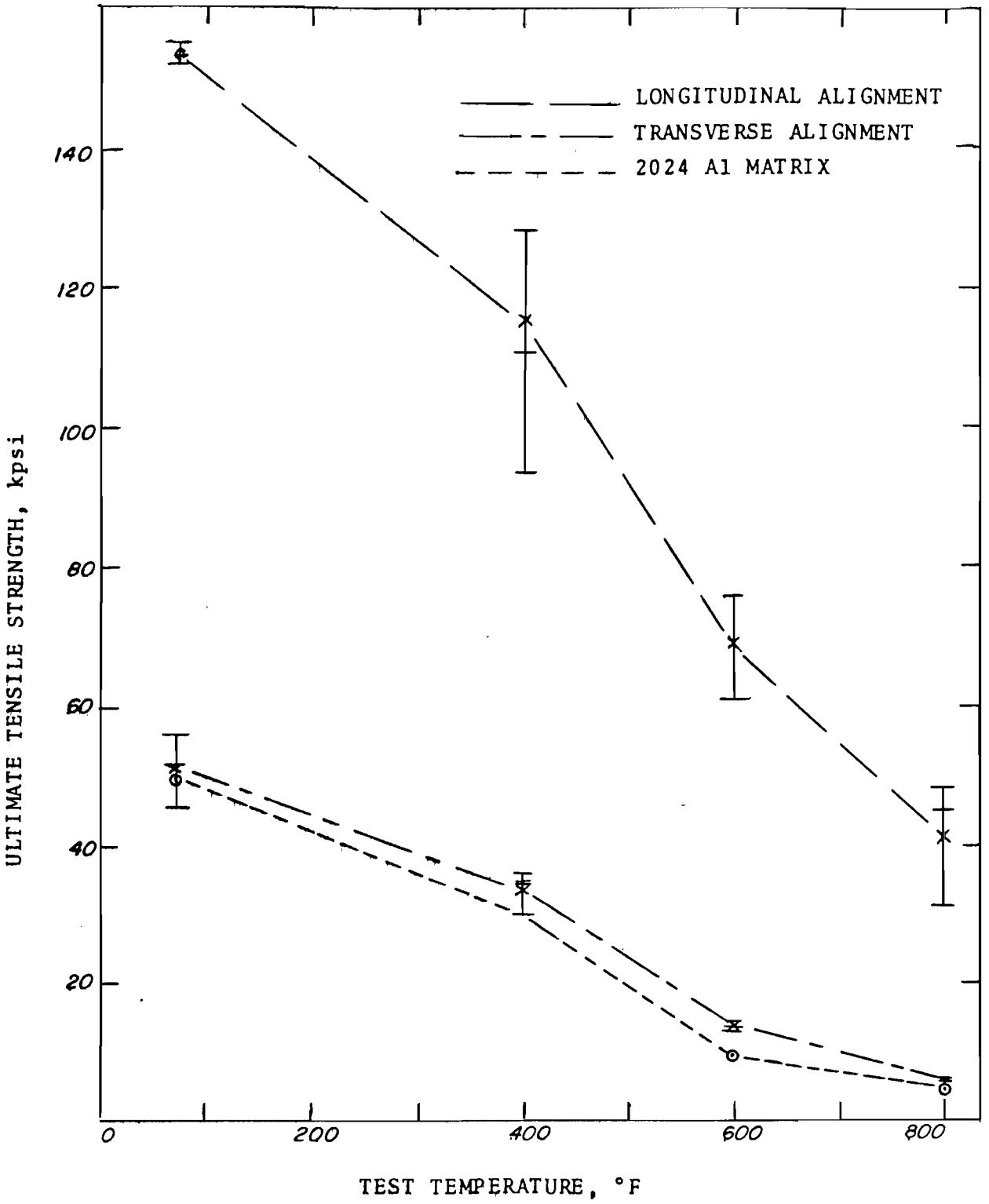

Figure 3. Effect of temperature on the longitudinal and transverse ultimate tensile strength of aligned 2024 Al/25 v/o β-SiC composites.

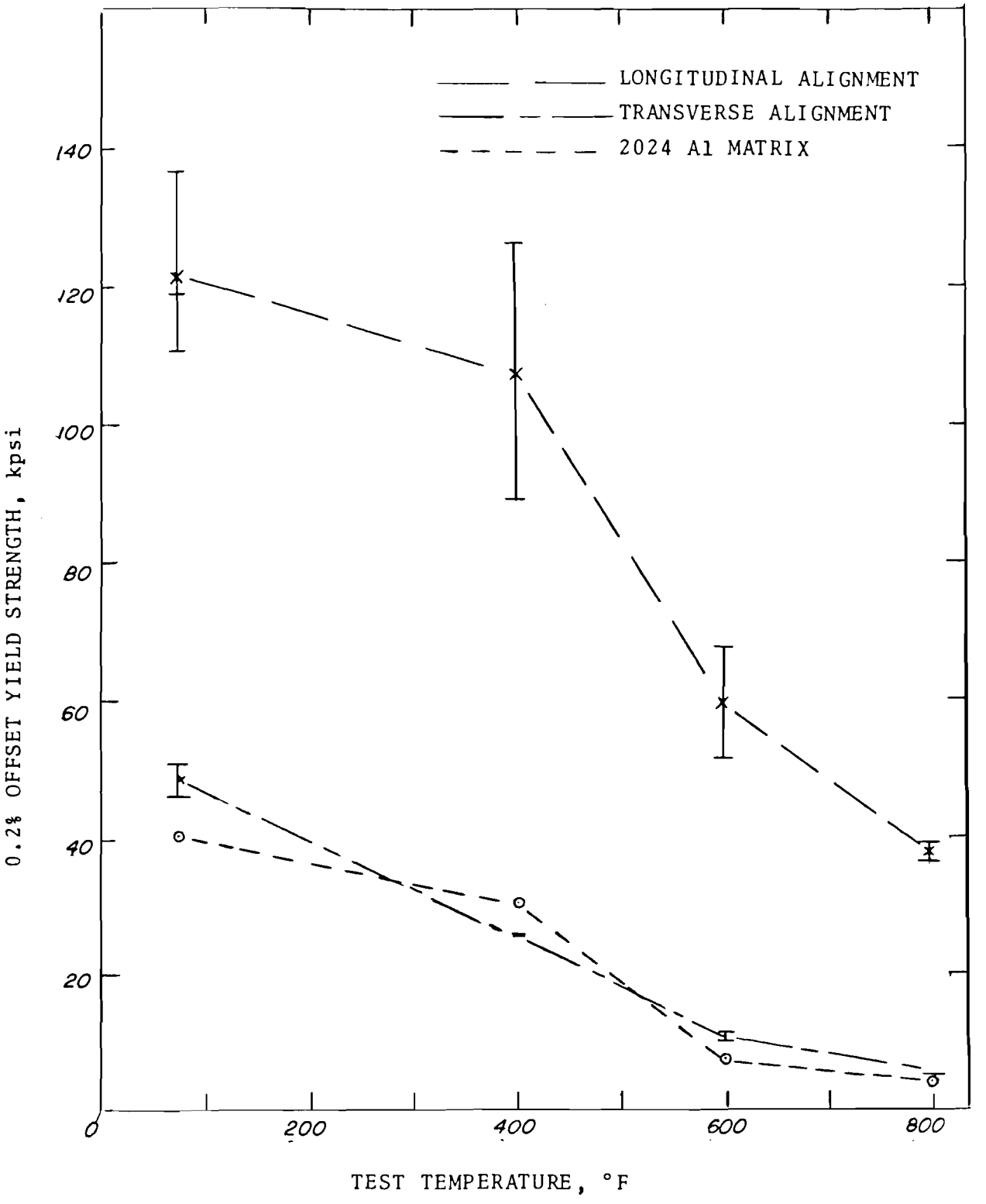

Figure 4. Effect of temperature on the longitudinal and transverse tensile yield strength of aligned 2024 Al/25 v/o β-SiC composites.

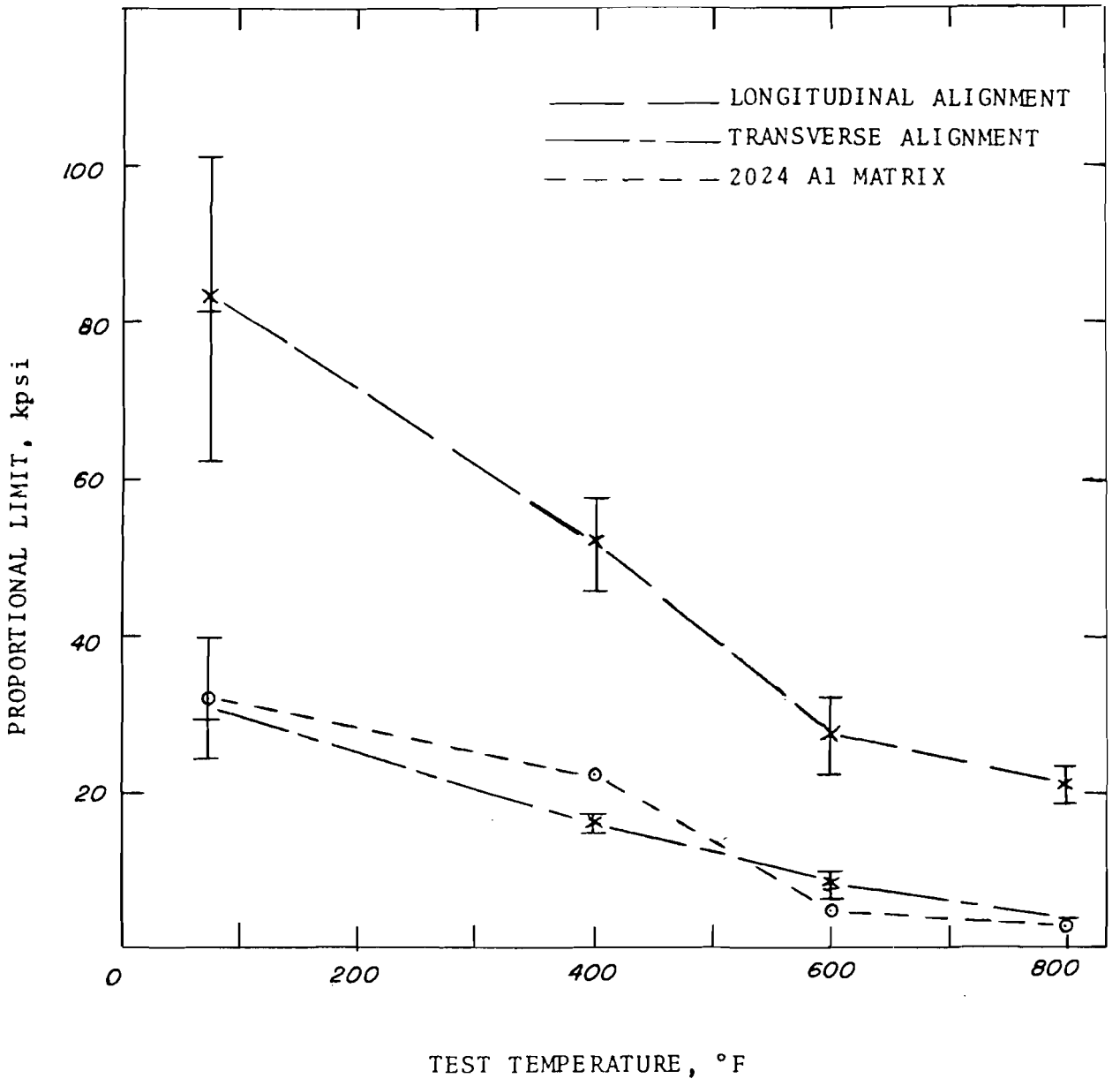

Figure 5. Effect of temperature on the longitudinal and transverse proportional limit of aligned 2024 Al/25 v/o β-SiC composites.

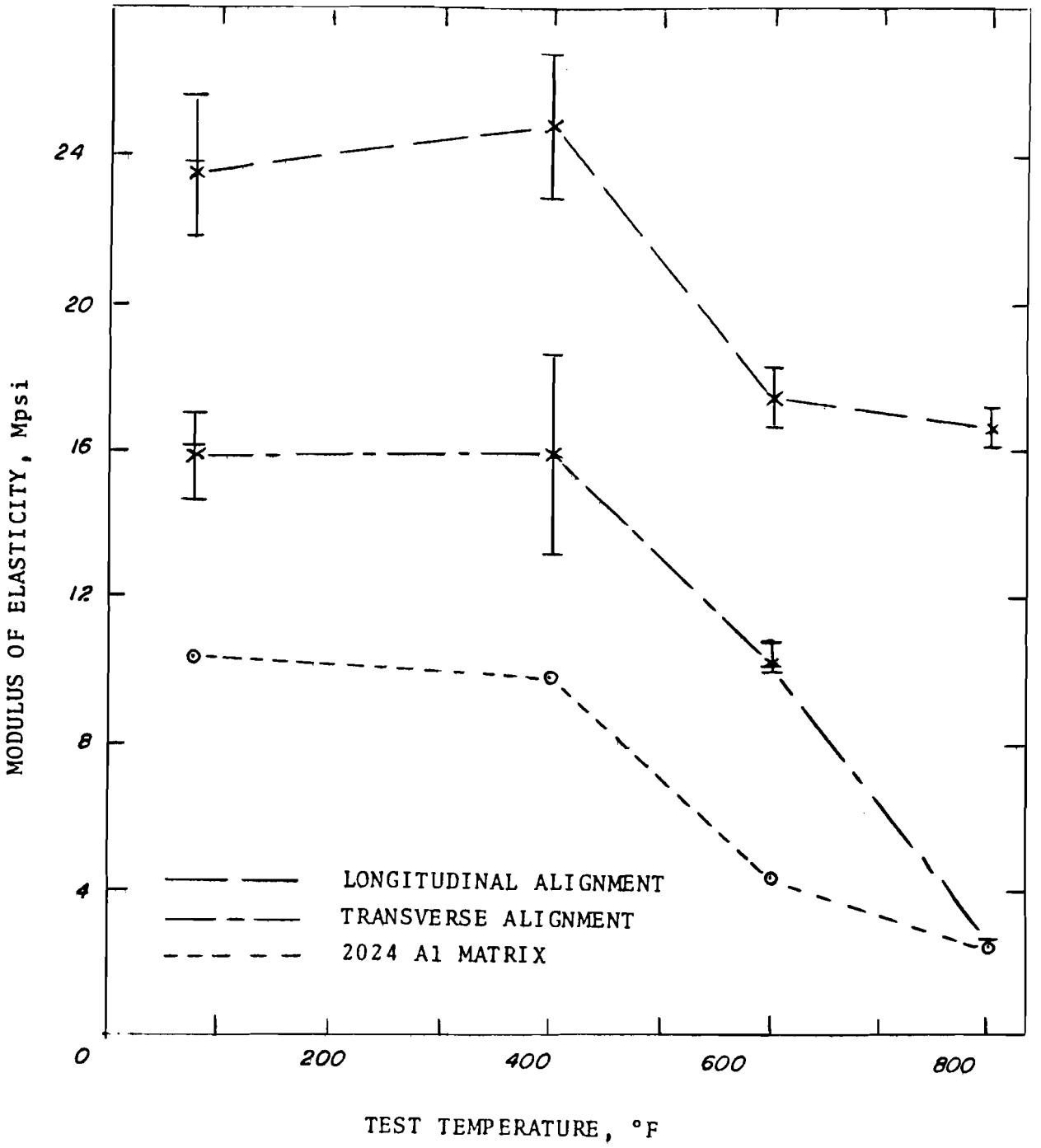

Figure 6. Effect of temperature on the longitudinal and transverse modulus of elasticity of aligned 2024 Al/25 v/o β-SiC composites.

of three at room temperature and nearly ten at 800°F, as can be seen from figure 3. Similar property increases were also realized for the tensile yield strength, proportional limit, and modulus of elasticity as is evident from figures 4 through 6.

For specimens in which the whiskers were aligned perpendicular to the tensile axis, all mentioned properties were slightly increased above the matrix properties except for the elastic modulus which was increased by approximately 50%. Most likely this increase is associated with the transversely aligned whiskers restricting the lateral strain imposed by the elongating specimen. The apparent decrease in proportional limit and tensile yield strength at the 400°F test temperature may have been related to temperature control problems that developed during testing.

Microscopic examination of the fracture faces revealed characteristics which were related to the whisker alignment and the test temperature. For the room temperature transverse test specimens the fracture faces appeared flat or slightly "V" shaped with the surface relatively smooth as shown in figure 7(a). Contrasting this is an 800°F transverse fracture face, figure 7(a), which is characterized by "islands" of whisker bundles torn free and bent 90 degrees which then failed with the whiskers axially aligned. The transverse specimens tested at 400 and 600°F appeared similar to the latter but with fewer islands at the lower temperature.

Similarly, the longitudinal specimens also were observed to have a characteristic appearance. In figure 7(b) the room temperature fracture face appears relatively flat and granular while the 800°F one has an irregular fibrous appearance. The 400 and 600°F fractures were very similar to the 75°F specimens. Examination of these fracture faces at higher magnification showed that whisker pullout was not evident in the 75 and 400°F longitudinal test specimens. Relatively few were noted in the 600°F specimens while a significant number were observed in the 800°F specimens as can be seen in figure 8. The protruding whisker lengths appeared to be similar for both temperature test specimens suggesting that failure was associated with the rapid loss of matrix shear strength in this temperature range.

Although the results obtained during testing of the specimens were significant, many factors were operating that could have influenced and possibly limited the results. For example, procedural difficulties developed during the early phase of testing concerning temperature control variation. While the temperature controller was capable of

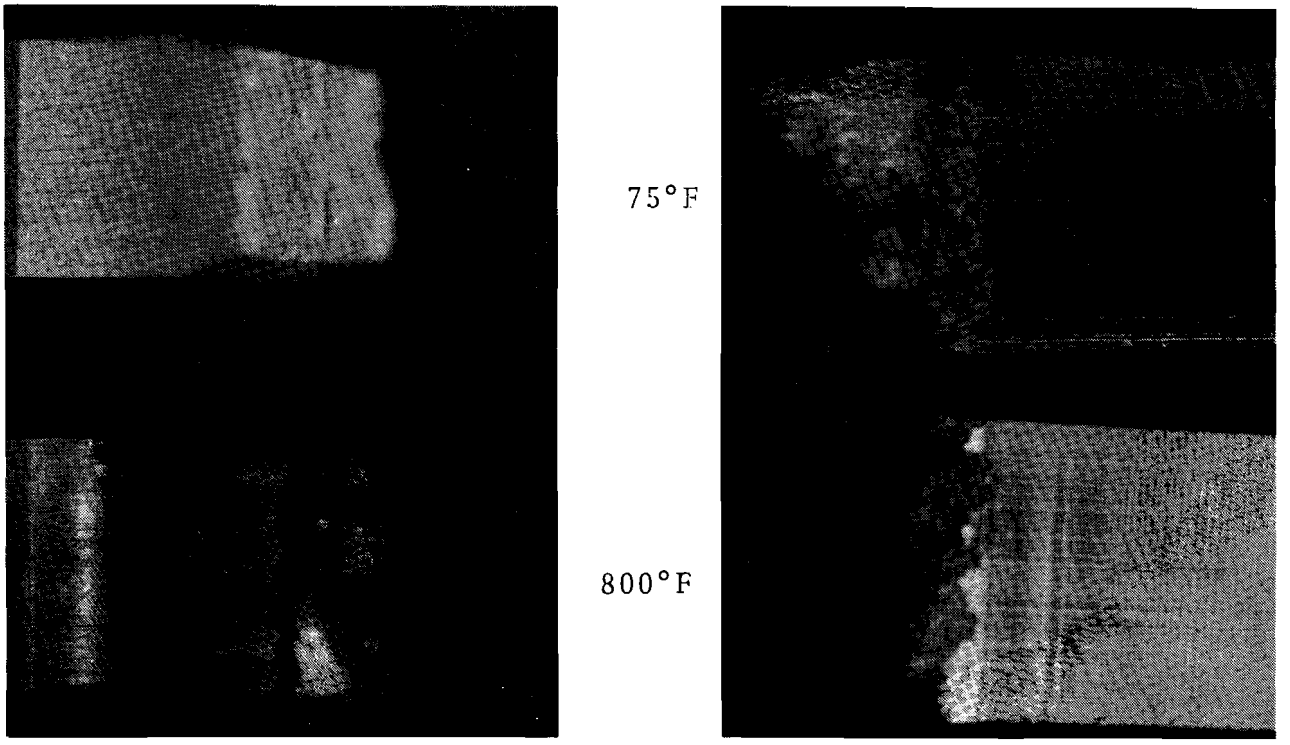

Figure 7.
Typical fracture appearance of specimens tested at 75°F and 800°F, X12: (a) transverse whisker alignment, (b) longitudinal whisker alignment.

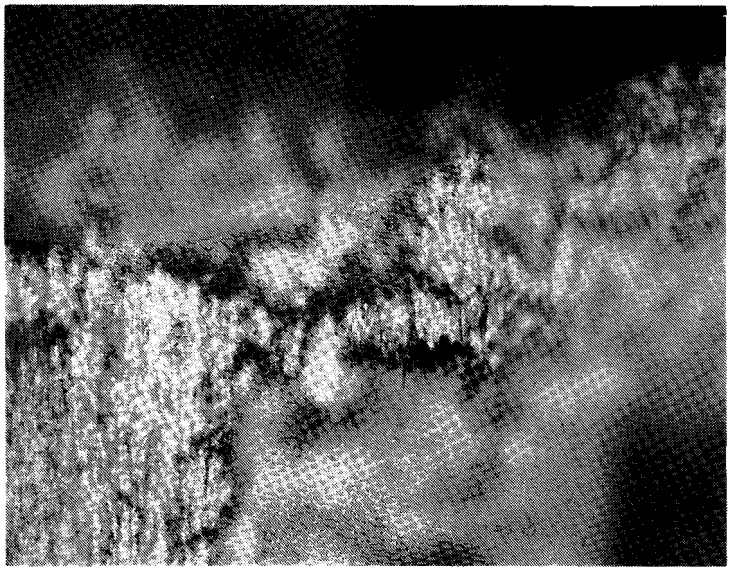

Figure 8.
Whisker pullout observed in fracture face of 800°F longitudinal specimens, (X50).

maintaining the furnace within ± 10°F, the temperature of the specimen was found to vary as much as 55°F with the top pin grip being hotter than the bottom because of the heat flow into the grips. By attaching the controlling thermocouple to the bottom grip and the recording thermocouple to the top grip, an average test temperature in the gage length appeared to be maintained at the lower test temperature. However, it appears that control of the high temperature was poorer with the upper grip being above 800°F. Shown in figure 9 are the three 800°F longitudinal test specimens. Deformation in the top grip at the shoulder pin area was common to all three specimens with the first specimen showing a shear failure in the top grip and deformation also present in the shoulder pin area of the lower grip. All three specimens failed either directly as a result of the top grip being hotter than the gage length or indirectly as a result of the specimen misaligning in the top grip.

Two additional problems are illustrated in figure 10; the center specimen involving extensometer problems and the other two specimens concerning preparation problems. Twelve specimens failed, through an impression imposed by one of the knife edges of the extensometer. Examination at a higher magnification as shown in figure 11 reveals the transition from smeared edge to granular fracture surface of a longitudinal specimen. Because of the weight of the extension arms their excessive movement during attachment and removal of the extensometer may have accentuated the damage to the specimen by the knife edges. To illustrate the specimen preparation problems, fifteen specimens failed in the proximity of a radius at one end of the gage length. This condition most likely occurred as a result of the difficulty of machining these specimens because of excessive wear of the carbide cutters. Because of these two problems total strain to failure could not be determined.

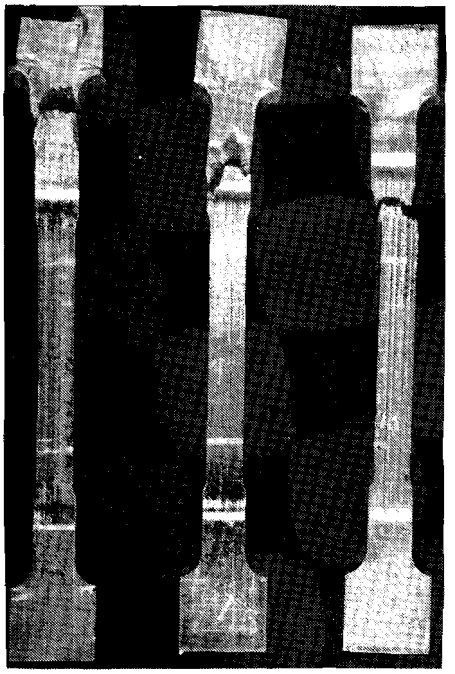

Figure 9.

Deformation in grip areas of 800°F longitudinal test specimens as a result of excessive temperature, (X2.5).

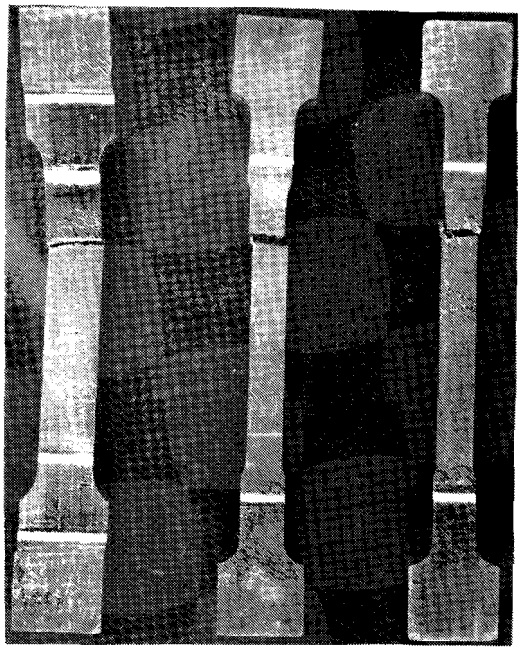

Figure 10.

Typical fracture locations which occurred as a result extensometer knife edge damage (center specimen) and tensile specimen preparation problems, (X2.5).

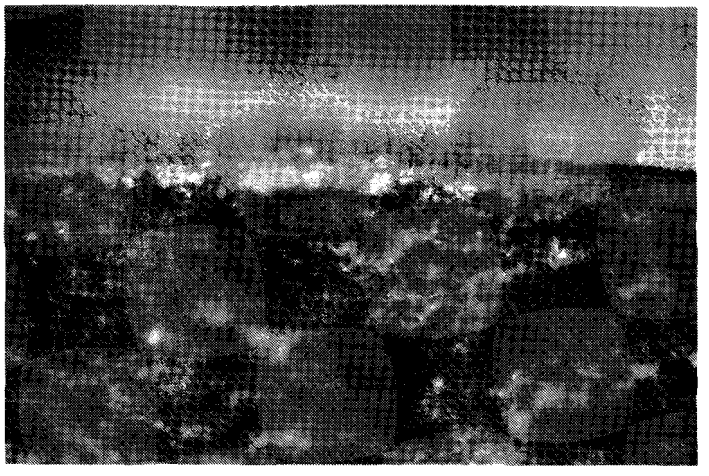

Figure 11.

Fracture face showing representative type of damage to specimens by knife edge of extensometer, (X75).

2. Ti-10V/SiC Composites Prepared by Activated Pressure Sintering

Before the initiation of mechanical testing of the Ti-10V/SiC composites, fibers were extracted as previously described in Section 3.2. These studies indicated that the elongation of filaments during pressing must be constrained to less than 1% to eliminate fracturing by thermal stressing.

Consolidation of composites for mechanical testing were accomplished as described in Section 2.2.2. Tensile test specimens were prepared from the consolidated samples in two stages. The primary objective was to prevent flexing of the sample during machining as a result of asymmetrical removal of material. In the first stage a fixture clamped the sample so that approximately equal amounts of material were removed from one half of the two opposite sides. By reclamping the sample in the roughed-out portion, the first stage of machining was completed without significant flexing of the sample. Finish machining was performed in the second stage without the use of the clamping fixture. In the resulting tensile test specimen, the strips of titanium alloy which were used as a backup support during consolidation became the grip portion of the specimen.

The results of the tensile testing, presented in Table IV, indicate that the composite specimens were inferior in strength to the unreinforced specimens. The influence of

TABLE IV

TENSILE TESTS OF Ti-10V/SiC FIBER COMPOSITE SAMPLES

Specimen Number	Specimen Type	Cross-Sectional Area, $in.^2$	Ultimate Tensile Strength, kpsi	Elastic Modulus, Mpsi	Strain to Fracture
Ti 10V 44	Unreinforced	0.00412	104.4	13.9	0.0078
Ti 10V 45	"	.00394	111.7	15.2	.0074
Ti 10V 46	"	.00426	126.8	16.7	.0079
Ti 10V 47	Composite	.00452	99.6	16.3	.0065
Ti 10V 48	"	.00384	112.0	17.5	.0072
Ti 10V 49	"	.00333	66.1	14.8	.0047

consolidating pressure is evident in the lower strength of composite specimen 49, to which the initial consolidating pressure was not applied. The fracture faces of the composite specimens revealed voids at the bottoms of the grooves, indicating incomplete consolidation. As voids were not observed in the areas above the fibers where strips of camphene saturated titanium alloy hydride power had been placed prior to consolidation, their presence at the bottoms of the grooves suggested that the grooved strips had not deformed around the fibers during the final consolidation step. The lack of deformation of the grooved strip suggested that the strips had become contaminated and embrittled during earlier consolidation. As this condition had not been observed in previous specimens, two additional composite samples were prepared using two grooved strips in each. The consolidation parameters were identical for these two composite samples except that the initial consolidating pressure was applied to only one. Both samples were sectioned longitudinally and examined metallographically. The same lack of deformation was noted in these samples which were known to be uncontaminated.

Since reaction has been observed in samples produced at higher temperatures or at this pressing temperature for longer periods, work was discontinued on this system.

3. Copper Coated Chopped Carbon Filament Al Alloy Composites

The initial results obtained from the previous investigation indicated that properties of Al alloy composites containing 40% chopped carbon filament might be significantly improved provided that fiber to fiber contact could be eliminated. The best properties, attained with Modmor fibers aligned in a 2024 Al matrix, yielded nearly a factor of three increase in the elastic modulus. Low tensile strengths were attributed to voids at numerous fiber to fiber contact points.

During the present contract several experiments were conducted in a limited effort to improve these properties. Since wetting of the carbon fibers was apparently sufficient by the 2024 Al alloy, a copper bearing alloy, to provide elastic reinforcement, coating of the carbon fibers with copper was considered as an possible solution since carbon to carbon contact would be eliminated. Another beneficial aspect gained was the increase in the density of the fiber by the coating. Previously, the fibers tended to segregate during mixing with the matrix powder since they were much less dense.

Attempts to produce a sample sufficiently dense, greater than 95%, for testing were unsuccessful. Because of the additional copper content, an excessive quantity of liquid was produced by reaction with the 2024 Al matrix during consolidation. The high liquid content resulted in voids at carbon to carbon contact points. Inconsistent results were obtained during the process of reestablishing the consolidation temperature at 548°C because of the inability to control the amount of copper in the system. Consequently, this effort was terminated because of the variable nature of the total surface area of the fibers and the coating process.

SECTION V
CONCLUSIONS

Consolidation of high volume fraction β-SiC/7178 Al alloy composites were unsuccessful. Voids present in the composites were probably caused by lack of penetration of the liquid into spaces created by slight misalignment of the whiskers. Consolidation of 7178 Al alloy composites by squeezing liquid from a low volume fraction composite into a reservoir may be a promising technique for preparing samples.

Significant increases in the mechanical properties of 25% β-SiC/2024 Al alloy composites were measured. Longitudinal tensile properties were increased three-fold times at room temperature and ten-fold at 800°F. Transverse properties were equal to or greater than the unreinforced matrix at all temperatures tested.

The SiC filament/Ti-10V composites prepared by activated pressure sintering did not yield materials which were stronger than the unreinforced matrix. Some question exists as to whether the Ti-10V hydride was contaminated during fabrication or whether full reduction of the hydride requires either higher temperatures or longer times than those utilized. If the latter is the case, the system is impractical since the reaction between the matrix and the filament is too severe.

SECTION VI
FUTURE WORK

A limiting factor in the utilization of continuous filament metal matrix composites is their low transverse tensile strength, which is associated with weakness in compression and shear. It is thought that a program which combines the technology of discontinuous reinforcement developed on this program with that of the continuous filament boron and Borsic(R) composites would be advantageous. By utilizing high strength, high modulus β-SiC whiskers as a matrix reinforcement in a continuous filament composite, increased transverse strengthening of continuous filaments composites could be achieved without the penalty of loss of specific strength from the utilization of metal wires such as stainless steel.

The objectives of such a program would be five fold:

(1) To improve the transverse strength of continuous filament metal matrix composites by off-axis reinforcement with β-SiC whiskers.

(2) To establish the volume fraction and orientation of the whiskers necessary to optimize the transverse strength.

(3) To characterize the composite so produced in terms of room temperature and elevated temperature mechanical properties.

(4) To investigate the feasibility of adapting fabrication techniques to a suitable technique for large scale use.

(5) To utilize fabrication processes which take advantage of the current state of the art in fabrication of B/Al components and are potentially compatible with available techniques for producing large sheets and cylindrical sections.

SECTION VII

TECHNOLOGICAL FORECAST FOR LIGHT METAL/DISCONTINUOUS FIBER REINFORCED COMPOSITES

1. Background

A discontinuous fiber composite usually contains a strong, brittle, aligned phase distributed in a softer matrix alloy. This discontinuous fiber reinforces the composite by carrying the load transferred by matrix deformation. The matrix prevents formation of a continuous brittle path through the composite, binds and protects the fibers, and transfers stress between the fibers.

2. Present Status

Reinforcement of light metal alloys with discontinuous fibers has presently been limited to aluminum, magnesium, and titanium alloys reinforced with chopped graphite fibers, sapphire, α-SiC, and β-SiC whiskers. The best aluminum alloy whisker composite, which was prepared under Navy sponsorship, has a compressive strength of 250,000 psi and elastic modulus in tension of 27.0×10^6 psi with 25 volume percent β-SiC whiskers.

During the past year, elevated temperature tensile properties have be en determined for the β-SiC/2024 Al composites. The composites have transverse tensile strengths at room temperature and elevated temperatures greater than that of the matrix. This is significant since the tensile strength of continuous filament composites are approximately 25% less in the transverse direction.

Work on the activated pressure sintering of SiC/Ti-10V composites indicates that the temperature required for complete densification is higher than can be tolerated in terms of filament/matrix interaction. Since the application of CeS as a barrier coating would be expensive in production and there is interaction be Ti-10V and the reinforcement, work has been discontinued on this system.

3. Forecast

The role of composites in future materials technology will be extensive. Continuous and discontinuous fiber reinforced composites will provide highly efficient special-purpose materials with properties that are not currently available in conventional materials. Metal matrices will be

increasingly employed for higher strength and higher temperature applications. Continuous fiber reinforced composites will be used in applications requiring highest strengths in which cost is not a limiting factor. Discontinuous fiber metal-matrix composites will become available for commercial application later than continuous fiber composites because their development funding has been more restricted. Discontinuous fiber metal-matrix composites may be fabricated into intricate shapes and are not limited by required lamination or winding processes peculiar to composites reinforced with continuous filaments. Discontinuous fiber reinforced composites will provide strength and stiffness and maintain these properties at elevated temperatures. Alternatively, discontinuous composites will be utilized as transverse reinforcement in continuous composites to improve off-axis properties.

It is anticipated that high strength graphite fibers and β-SiC whiskers, which are several times more expensive at present but under intensive commercial development, will be the primary discontinuous fibers used for reinforcement. The mechanical properties of composites as anticipated in composites of the future are shown as a function of time in figure 12. The values shown are those for the best aluminum alloys reinforced by currently available whiskers, which may approach theoretical maximum strength values, or high strength fibers of graphite or other polycrystalline materials that will become available. It is interesting to note that points (1) (2) and (3) which are marked on the graphs represent two composites that have been produced since the preparation of that section of the property forecast curves. The strength and modulus of material produced have exceeded the predicted values up to 1972. The anticipated increased uses of these materials, represented by increasing production and decreasing cost, are similarly extended in figure 13 from previous forecast curves. The cost reductions and production increases have not occurred for these materials as rapidly as predicted.

4. Operational Implications

With improvement in fiber quality, lower cost, and continued progress in the development of fabrication techniques, discontinuous fiber light-metal composites are expected to be utilized in numerous applications requiring materials tailored to the job. Parts of complex configuration that require high stiffness/density and strength/density ratios are applications of immediate interest for these composites. Selective reinforcement will make these materials even more effective in the future for such possible applications as bearing surfaces, multi-hardness, and erosion-resistant components.

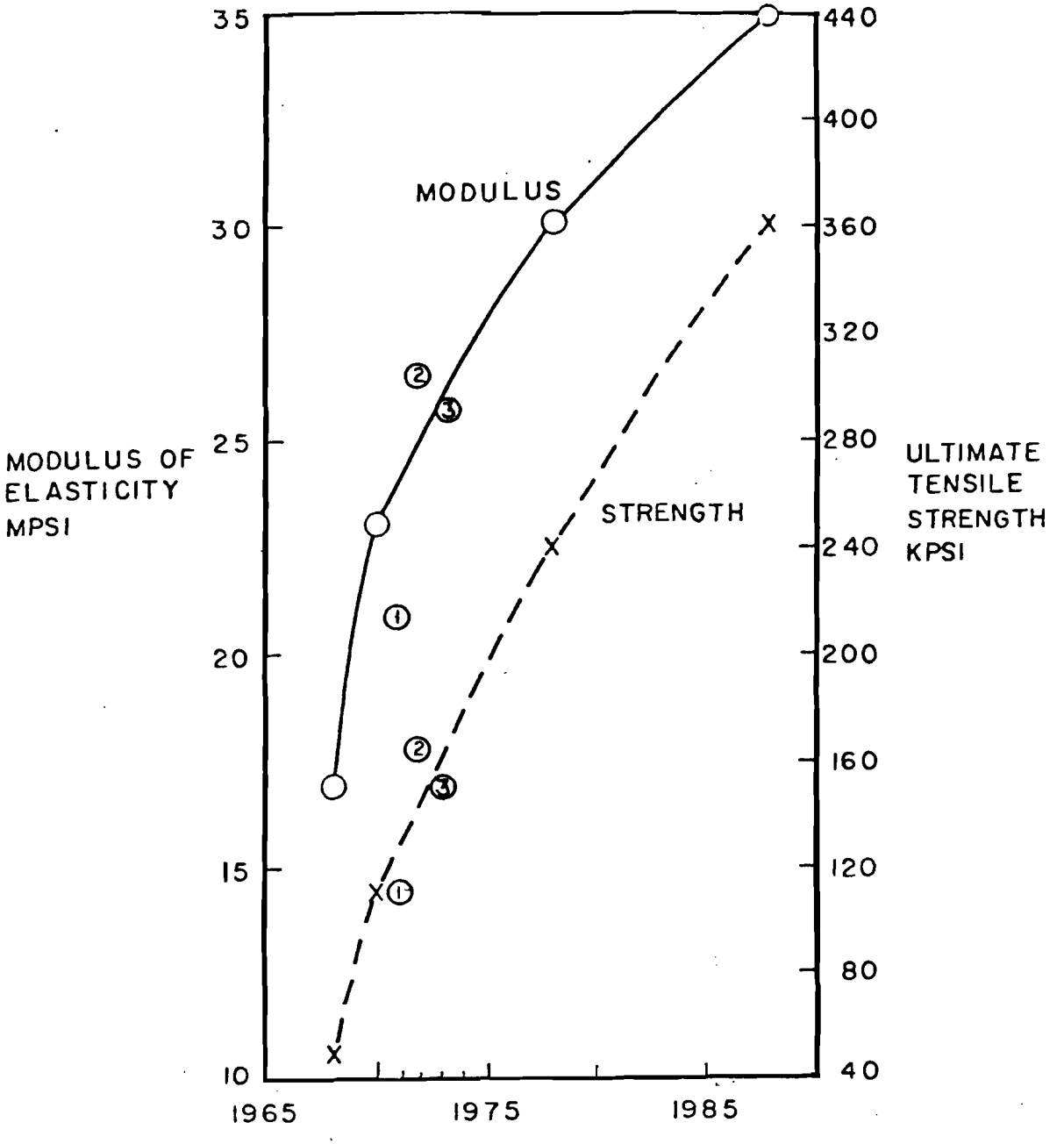

Figure 12. Predicted improvement in mechanical properties of 25 v/o discontinuous fiber light metal composites through 1990.

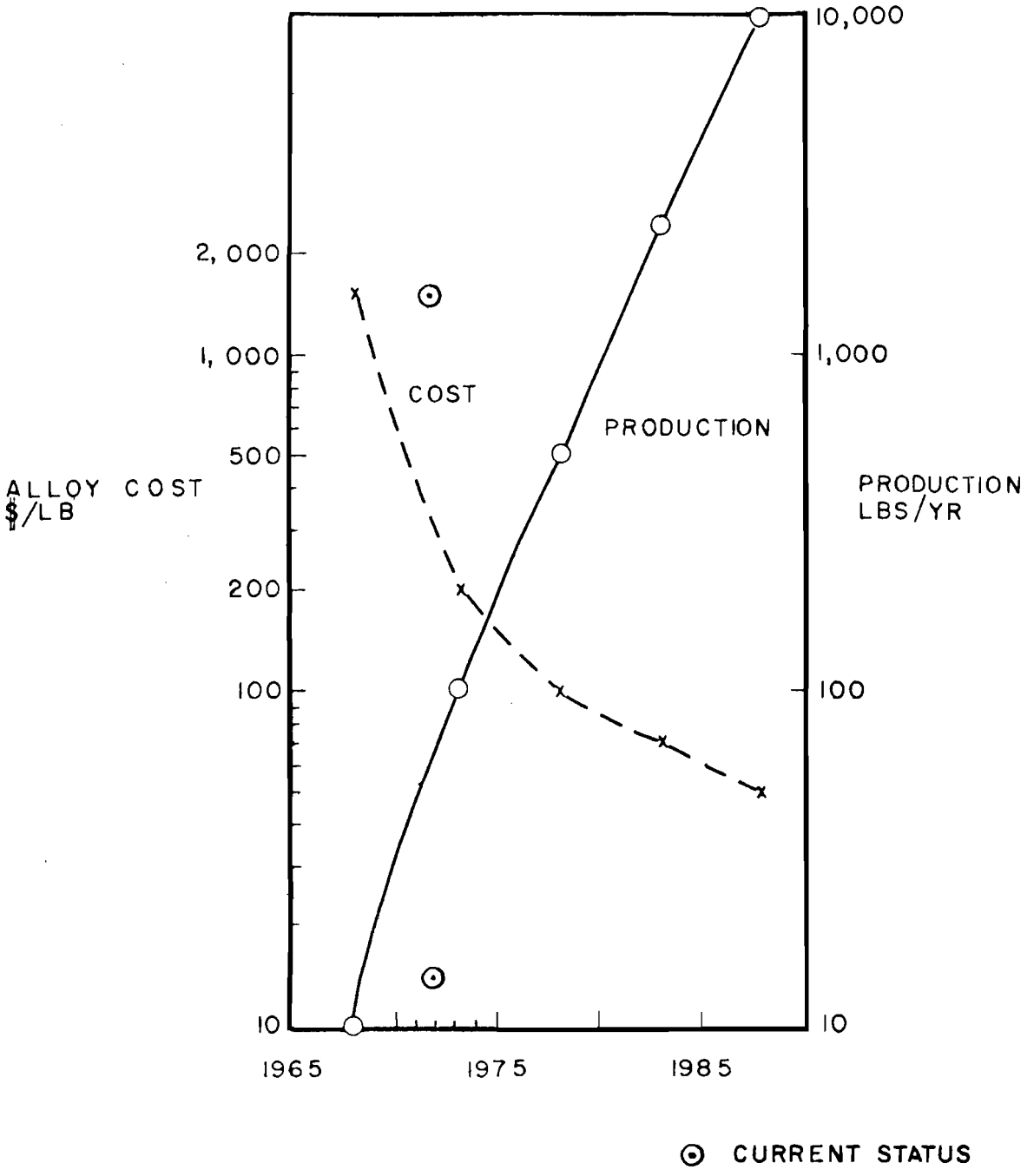

Figure 13. Predicted production and cost of discontinuous fiber reinforced composites through 1990.

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Titanium Hydride
Titanium Composites
Silicon Carbide
Activated Pressure Sintering
Ti-V Alloys
Graphite Fibers

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Discontinuous β-SiC whiskers and chopped C filaments were used as a reinforcement for strengthening 7178 and 2024 Al alloys. The C fibers were coated with Cu to improve wetting. Tensile properties of a 25 v/o β-SiC 2024 Al composite were increased three times over the matrix strength at room temperature and ten times at 800°F. Tensile properties in the transverse direction were at least equal to the matrix strength at temperatures up to 800°F.

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(20) Ti-10V composites reinforced with SiC filaments were consolidated by activated pressure sintering. The samples produced were not fully dense. Consolidation temperature or time at temperature could not be increased since the reaction between the matrix and filaments would be too severe. Work on this system was terminated.